Family list
1 family member for: **JP63131494**
Derived from 1 application

1 **THIN FILM EL DEVICE AND MANUFACTURE OF THE SAME**
Inventor: SHIMIZU YASUMOTO **Applicant:** HOYA CORP
EC: **IPC:** ***H05B33/04***; *H01L51/52*; **H05B33/04** (+2)
Publication info: JP63131494 A - 1988-06-03

Data supplied from the ***esp@cenet*** database - Worldwide

⑲日本国特許庁（JP）　⑪特許出願公開

⑫公開特許公報（A）　昭63－131494

⑤1Int.Cl.⁴　識別記号　庁内整理番号　⑬公開　昭和63年(1988)6月3日
H 05 B 33/04　6744－3K

審査請求　未請求　発明の数 2　（全6頁）

⑤4発明の名称　薄膜ＥＬ素子およびその製造方法

⑳特　願　昭61－275150
㉒出　願　昭61(1986)11月20日

⑦2発明者　清水　安元　東京都新宿区中落合２丁目７番５号　ホーヤ株式会社内
⑦1出願人　ホーヤ株式会社　東京都新宿区中落合２丁目７番５号
⑦4代理人　弁理士　山川　政樹　外２名

明　細　書

1. 発明の名称

薄膜EL素子およびその製造方法

2. 特許請求の範囲

(1) 透明基板と、この透明基板上に形成された透明電極、誘電体層、EL発光層および背面電極を含むEL薄膜層と、このEL薄膜層を覆つて透明基板上に固着されたキャップとを有する薄膜EL素子において、キャップは、接着剤注入口を備えるとともに内面に吸湿性材料を固定し、かつ上記接着剤注入口は熱硬化形樹脂接着剤により封じてなることを特徴とする薄膜EL素子。

(2) キャップ内面に吸湿性材料を分散させた接着剤を塗布し硬化させて吸湿性材料を固定する工程と、このキャップを、透明電極、誘電体層、EL発光層および背面電極からなるEL薄膜層を設けた透明基板に固着する工程と、これらキャップおよび透明基板を加熱した状態で、キャップに設けた接着剤注入口に熱硬化形樹脂接着剤を注入し硬化させて封止する工程とを含むことを特徴とする薄膜EL素子の製造方法。

3. 発明の詳細な説明

〔産業上の利用分野〕

本発明は、平面形ディスプレイ・デバイスとして、コンピュータシステムの端末機器その他の表示装置に、静止画像や動画像の表示手段として利用される薄膜EL素子およびその製造方法、特に封じ方法に関する。

〔従来の技術〕

従来この種の薄膜EL（エレクトロルミネセンス）素子は、例えば透明なガラス基板上に、In_2O_3，SnO_2 等からなる透明電極を配列し、次に Y_2O_3，Ta_2O_5 等からなる第１の誘電体層、発光中心として0.1～2wt%のMnをドープした ZnS 等からなるEL発光層および第２の誘電体層を順次積層した後、Al，Ta，Mo等からなる背面電極を配列することによつて形成されていた。透明基板側から見て透明電極と背面電極とが交差する領域がパネルの１絵素に相当し、両電極間に交流電圧を印加することにより、Mn発光中心より

(19) Japan Patent Office (JP)

(12) Publication of Unexamined Patent Application (A)

(11) Publication Number: Japanese Published Patent Application No.: S63-131494

(51) Int.Cl.[4]	Identification Symbol	JPO File No.
H 05 B 33/04		6744-3K

(43) Date of Publication: June 3rd, 1988

Request for examination: Not made

Number of Inventions: 2 (6 pages in total)

(54) Title of the Invention: Thin Film EL Element and Manufacturing Method Thereof

(21) Patent Application No.: S61-275150

(22) Date of Filing of Patent Application: November 20th, 1986

(72) Inventor: Yasumoto SHIMIZU

C/o: Hoya Corporation

2-7-5 Naka-Ochiai, Shinjuku-ku, Tokyo

(71) Applicant: Hoya Corporation

2-7-5 Naka-Ochiai, Shinjuku-ku, Tokyo

(74) Representative: Patent Attorney, Masaki YAMAKAWA, and two others

Specification

1. Title of the Invention

Thin Film EL Element and Manufacturing Method Thereof

2. Scope of Patent Claims

(1) A thin film EL element characterized by comprising: a transparent substrate; a transparent electrode formed over this transparent substrate; a dielectric layer; an EL thin film layer which contains an EL light-emitting layer and a back surface electrode; and a cap which covers this EL thin film layer and which is attached to the transparent substrate; wherein the cap, along with being equipped with an adhesive injection port, is fixed to an inner surface using a hygroscopic material; and the adhesive injection port is sealed with a thermosetting resin adhesive.

(2) A method for manufacturing a thin film EL element characterized by comprising

the steps of: a step for applying an adhesive diffused throughout a hygroscopic material to an inner surface of a cap and affixing the hardened hygroscopic material to the inner surface of the cap; a step for attaching this cap to a transparent substrate provided with an EL thin film layer composed of a transparent electrode, a dielectric layer, an EL light-emitting layer, and a back surface electrode; and a step for injecting a thermosetting resin adhesive into the thin film EL element through an adhesive injection port provided in the cap, hardening the thermosetting resin adhesive, and sealing the cap and the transparent electrode, all while this cap and this transparent electrode are being heated.

3. Detailed Description of the Invention

[Field of Industrial Application]

The present invention relates to a thin film EL element used as a displaying means for still images and video in a computer system terminal device as well as other display devices, for a planar display device and to a manufacturing method thereof, in particular, to a sealing method thereof.

[Related Art]

Traditionally, this kind of thin film EL (Electroluminescence) element was formed, for example, in the following way: a transparent electrode made from In_2O_3, SnO_2, or the like is placed over a transparent glass substrate; next, a first dielectric layer made from Y_2O_3, Ta_2O_5, or the like, an EL light-emitting layer made from ZnS or the like doped with from 0.1 wt% to 2 wt% of Mn acting as a luminescent center, and a second dielectric layer are stacked, in order; and, after that, a back surface electrode made from Al, Ta, Mo, or the like is placed over the stacked layers. Viewed from the transparent substrate side, a region where the transparent electrode and the back surface electrode intersect corresponds to one picture element of a panel, and by application of an alternating current between both electrodes, emission of orange light by the Mn luminescent center is exhibited.

In this kind of EL element, a thin film multilayer object (hereinafter referred to as an EL thin film layer) from the transparent electrode formed over the transparent substrate to the back surface electrode is easily affected by ambient air, in particular, by

moisture, and even if a tiny bit of moisture from the atmosphere is adsorbed by an EL thin film layer, the moisture penetrates into pinholes and the like in the dielectric layer and the like, whereby the resistance of that portion is lowered. As a result, too much current flows, a localized portion becomes heated, the thin film is separated from the substrate, dielectric breakdown occurs, and the life of the element comes to be shortened. In addition, the penetrated moisture reaches the EL light-emitting layer, and because the light-emitting layer is extremely weak against water, the light-emitting layer deteriorates, and the life of the element comes to be shortened.

In order that the thin film EL element be protected from this kind of moisture, in the past, the following measures were taken: **(1)** an inorganic or organic coating was firmly attached to cover the entire EL thin film layer; **(2)** the EL thin film layer was covered by a glass cap or the like; **(3)** the glass cap in (2) was degassed so that the inside of the glass cap was drawn down to vacuum; **(4)** the inside of the glass cap in (2) was filled with an insulating liquid, such as with silicon oil or the like; and **(5)** in addition to what was performed in (4), furthermore, a sheet to which a silica gel, which acts as a moisture absorber, has been applied is attached to the inner walls of the cap or the like.

[Problems to be Solved by the Invention]

In each of the above moisture-prevention measures, each one has drawbacks in production in addition to advantages. First of all, in ①, having to dissipate heat from the EL element is inconvenient, and, in some cases, separation of layers occurs due to an increase in stress. Next, in ②, there is not enough of an effect. In addition, in each of ③, ④, and ⑤, there are difficulties in production; furthermore, in ③, in particular, because there is a difference in pressure between external atmospheric pressure and the pressure inside the cap, there are problems with the strength of the element, as well.

[Means for Solving the Problems]

In a thin film EL element of the present invention, a cap, to the inner surface of which a hygroscopic material is applied, is used, and an adhesive injection port provided in the cap is sealed with a thermosetting resin adhesive.

In addition, in a manufacturing method of the present invention, the following steps are provided: a step for applying an adhesive diffused throughout a hygroscopic material to an inner surface of a cap and affixing the hardened hygroscopic material to the inner surface of the cap; a step for attaching this cap to a transparent substrate provided with an EL thin film layer; and a step for injecting a thermosetting resin adhesive into the thin film EL element layer through an adhesive injection port provided in the cap, hardening the thermosetting resin adhesive, and sealing the cap and the transparent substrate, all while this cap and this transparent substrate are being heated.

[Operation]

By heating of the cap which is arranged over the transparent substrate, moisture remaining inside the cap is removed through the adhesive injection port while the penetration of moisture from the external atmosphere is prevented. After the cap has been sealed, even if a tiny bit of moisture is left remaining inside the cap, that moisture is absorbed by the hygroscopic material affixed to the inside of the cap.

[Embodiments]

FIGS. 2(a) and 2(b) show an EL thin film layer formed over a transparent substrate; FIG. 2(a) is a plane-view diagram, and FIG. 2(b) is a cross-sectional view taken along b-b in FIG. 2(a). Next, this formation method will be described.

First, over a transparent substrate 1 made from aluminosilicate glass (manufactured by HOYA Corp., NA40; dimensions of 100 mm x 100 mm x 3 mm), indium oxide into which a tin oxide is mixed is set as a raw material, and a number of strip-shaped transparent electrodes 2 are formed by a vacuum deposition method. At this time, drawn-out portions of the transparent electrodes 2 are made to extend toward the periphery of the substrate in an alternating fashion. Next, by a similar manufacturing method, a first dielectric layer 3 (film thickness of 3000 Å) with yttrium oxide (Y_2O_3) set as the raw material; an EL light-emitting layer 4 (film thickness of 6000 Å) with sintered pellets of ZnS:Mn, doped with 0.5 wt% of manganese (Mn) acting as the active material, set as the raw material; and a second dielectric layer 5 (film thickness of 3000 Å) with the same material as was used for the first dielectric layer 3 set as the raw material are formed in order. Then, over the second dielectric

layer 5, by a similar manufacturing method, with aluminum (Al) set as the raw material, a number of strip-shaped back surface electrodes 6 (film thickness of 3000 Å) are formed in a direction in which the back surface electrodes 6 intersect with the transparent electrodes 2 in an alternating fashion. Drawn-out portions of these back surface electrodes 6 also extend toward the periphery of the substrate in an alternating fashion. An EL thin film layer 7 is formed of both electrodes minus these drawn-out portions as well as the EL light-emitting layer interposed therebetween and the first and second dielectric layers.

FIGS. 3(a) to 3(c) are diagrams showing a cap used and affixed to this transparent substrate 1; FIG. 3(a) is a plane-view diagram, Fig. 3(b) is a cross-sectional view taken along b-b in FIG. 3(a), and FIG. 3(c) is a perspective diagram (however, it is a reflected image). A cap 8 is made from aluminosilicate glass, the same raw material from which the transparent substrate 7 is made. The dimensions (unit: mm) for each part are as shown in FIGS. 2(a) and 2(b); the cap generally has a box shape with an approximately square bottom wall and four side walls, but in the open terminal, a rectangular notch 9 is formed. As will be described below, this notch 9 forms an adhesive injection port when the cap 8 is affixed to the transparent substrate 5 [sic].

Next, using FIGS. 1(a) to 1(c), a step for affixing the cap 8 to the transparent substrate 1 to seal the transparent substrate 1 in this manner will be explained.

At first, the cap 8 is preheated at from 100 °C to 200 °C (in the present embodiment, at 150 °C) for an hour or more (in the present embodiment, for two hours), and moisture adsorbed on the surface is adequately removed. Next, a paste dispersed throughout an epoxy-based adhesive formed of thermally cured fine particles of diphosphorus pentoxide (P_2O_5) is prepared; after the same paste is applied to the inner surface, which will come to face the EL thin film layer 7, of the bottom wall of the cap 8 at a thickness of from approximately 200 μm to 300 μm by a screen printing method, the cap 8 is placed on a hot plate or the like of a heater and heated at from 100 °C to 200 °C (at 150 °C in the present embodiment). Herewith, a hygroscopic effect layer 12 affixed to fine particles 11 of diphosphorus pentoxide solidified in an adhesive layer 10 is obtained (FIG. 1(a)).

Meanwhile, the transparent substrate 1 forming the EL thin film layer 7 is preheated at from 100 °C to 200 °C (at 120 °C in the present embodiment) for an hour or more (for three hours in the present embodiment), and moisture adsorbed on the surface and absorbed inside is adequately removed. While this is being continuously heated, over this transparent substrate 1, the aforementioned cap 8 is placed in a predetermined location so as to cover the EL thin film layer 7. At this time, on the surface of the open end of the cap 8, not including the notch 9, by application of a thermosetting epoxy-based adhesive in advance, the cap 8 is affixed onto the transparent substrate 1 by a solidified adhesive joint 13 formed from this hardened adhesive (FIG. 1(b)).

Next, after this transparent substrate 1 and this cap 8 are intermittently heated for a while, in that condition, a fresh epoxy-based adhesive is injected into the cap 8 through the notch 9 of the cap by a syringe or the like, and heating is continued for a predetermined length of time (for one hour in the present embodiment). Consequently, the notch 9 is sealed with a solidified adhesive joint 14 (FIG. 1(c)).

At the time of sealing, by preheating of each of the cap 8 and the transparent substrate 1 over which the EL thin film layer 7 is formed, moisture adsorbed on the surfaces of these and moisture absorbed into the EL thin film layer 7 can be removed. By further heating with these in a state of being stacked together, moisture remaining inside can be removed through the notch 9 in the cap 8, and penetration of moisture from the external atmosphere can be prevented. Furthermore, even if a small amount of moisture remains inside after sealing has been completed, because this moisture is absorbed by the fine particles 11 of the diphosphorus pentoxide, adverse effects, such as deterioration of the EL thin film layer 7, can be prevented from affecting the light-emitting display. The hygroscopic effect by this diphosphorus pentoxide, from being heated when sealing is performed, is further increased. In addition, from the fact that the adhesive solidifying the fine particles 11 of this diphosphorus pentoxide is a thermosetting type, deterioration does not occur due to heating during the sealing step.

In the present embodiment, the material for both the transparent substrate 1 and the cap 8 is set to be aluminosilicate glass; however, it is not limited to this. For

example, a ceramic or plastic material may be used, but, preferably, using a glass with an equivalent coefficient of thermal expansion is better. For this kind of glass, for example, a multi-component glass, such as soda lime glass or the like, may be used, or quartz glass may be used.

In addition, the thermosetting resin adhesive is not limited to an epoxy resin adhesive, and other resins, such as a silicone resin or the like, that have resistance to heat and moisture may be used.

Furthermore, for the hygroscopic material, in addition to diphosphorus pentoxide, silica gel, magnesium chlorate ($Mg(ClO_3)_2$), aluminum oxide (Al_2O_3), calcium oxide (CaO), or the like may be used, as well.

It is to be noted that, instead of the rectangular notch 9, a notch such as a V-shaped notch, a U-shaped notch, or the like or a through-hole such as a circular tube-shaped hole, an angular tube-shaped hole, or the like may be used; the number of notches or through-holes is not limited to one pair, and at least one is used. Moreover, if the adhesive injection port is a through-hole-shaped adhesive injection port, the location is not limited to sidewalls of the cap 8, and the adhesive injection port may also be provided in the bottom portion.

In addition, in the above-described embodiment, a cap is used in which the bottom portion and the sidewalls are formed altogether, but these may be formed separately and combined together. In FIGS. 4(a) to 4(c), that example is shown. FIG. 4(a) is a plane view diagram, FIG. 4(b) is a cross-sectional view diagram taken along b-b, and FIG. 4(c) is a perspective view diagram.

A cap 15 of the present embodiment has a structure in which, over one main surface of a plate-like cover 15A made of the same aluminosilicate glass as the transparent substrate 1, two U-shaped, slot-like spacers 15B are arranged facing each other. A thermosetting epoxy-based adhesive is applied to the plate-like cover 15A, and the spacers 15B are formed and affixed to the plate-like cover 15A by thermal hardening in an oven or the like at 100 °C. In that case, the two spacers 15B are divided from each other at a predetermined distance L = 0.3 mm to 10 mm (3 mm in the present embodiment) only on the open side of the U shape, and slits 16 are formed. It

is to be noted that the area within each slot of the two spacers has a size encircling the side surfaces of the EL thin film layer 7. In addition, for the dimension of that width W, 0.5 mm to 5 mm (2 mm in the present embodiment) is suitable. The dimension of the height H should be greater than the entire film thickness of the EL thin film layer 7 except for the transparent electrode 2 combined with the thickness of the solidified adsorbent layer to be described later (normally, from 20 μm to 3000 μm; from 200 μm to 300 μm in the present invention) as much as possible and was set to be 1000 μm in the present invention.

Next, using FIGS. 5(a) and 5(b), 6(a) and 6(b), 7(a) and 7(b), and 8(a) and 8(b), a step for affixing this kind of cap 15 to the transparent substrate 1 and sealing it will be explained. It is to be noted that the (a) of each diagram corresponds to a cross-sectional view of FIG. 4(b) and the (b) of each diagram corresponds to a side view of FIG. 4(b).

First, the cap 15 is preheated at from 100 °C to 200 °C (120 °C in the present embodiment) for one hour or more (for two hours in the present embodiment), and moisture adsorbed on the surface and absorbed inside is adequately removed. Next, a paste in which fine particles of diphosphorus pentoxide (P_2O_5) are dispersed throughout a thermosetting epoxy-based adhesive is applied at a thickness of from 20 μm to 3000 μm (from 200 μm to 300 μm in the present embodiment) to the inner surface of the plate-like cover 15A that faces the EL thin film layer 7 by a method similar to the one used in the embodiment for FIGS. 1(a) to 1(c) and made to harden, and the hygroscopic effect layer 12 made up of the hardened adhesive layer 10 of the fine particles 11 of diphosphorus pentoxide (P_2O_5) is obtained (FIGS. 5(a) and 5(b)).

On the other hand, the transparent substrate 1 forming the EL thin film layer 7 is also heated in a way similar to that of the embodiment of FIGS. 1(a) to 1(c), and after enough moisture is removed, while being continuously heated at from 100 °C to 200 °C (150 °C in the present embodiment), the cap 15 is arranged in a predetermined location so as to cover the EL thin film 7 (FIGS. 6(a) and 6(b)).

Next, into a recess 20 including an external surface 17 of the spacer 15B and oppositely faced surfaces 18 and 19 of the plate-like cover 15A and the transparent

substrate 1, a new epoxy-based adhesive is injected with a syringe or the like, and heating is continued for a predetermined length of time (for one hour in the present embodiment). Herewith, the epoxy-based adhesive hardens, and a solidified adhesive joint 21 is formed (FIGS. 7(a) and 7(b)). The solidified adhesive joint 21 is affixed to external surfaces of the transparent substrate 1, the plate-like cover 15A, and the spacer 15B, and these are securely joined together. In the drawings, an exaggerated view of this is drawn; because the dimension of the height (the same as the dimension of the height of the spacer 15B) H, as given above, does not exceed 1000 μm (1 mm), surface tension between the transparent substrate 1 and the plate-like cover 15A is obtained and the adhesive can be easily injected into the element. It is to be noted that the adhesive is not injected through the slit 16 at this time; an internal space is connected to the external atmosphere by the slit 16.

In this condition, after heating of the transparent substrate 1 and the cap 15 is continued for a while, a new epoxy-based adhesive is inserted, in advance, through the slits 16 with a syringe or the like, and heating is continued for a predetermined length of time (for 20 minutes in the present embodiment). Herewith, the slits 16 are sealed with a solidified adhesive joint 22 (FIGS. 8(a) and 8(b)).

In the present embodiment, the materials of the transparent substrate 1, the plate-like cover 15A, the thermosetting resin adhesive, the hygroscopic material, and the like not being limited to the materials described above is similar to that of the case of the embodiment of FIGS. 1(a) to 1(c).

Moreover, in each embodiment, the hygroscopic material is fixed only to the inner surface of the bottom portion of the cap (in the embodiment for FIGS. 5(a) and 5(b), to the inner surface of the plate-like cover 15A) but may be fixed to the inner surfaces of the sidewalls of the cap (in the embodiment for FIGS. 5(a) and 5(b), to the inner surface of the spacers 15B); of course, if the hygroscopic material is solidified over the entire inner surface of the cap, that hygroscopic effect can be increased.

It is to be noted that a structure is shown in which the EL light-emitting layer 4 is interposed between the first and second dielectric layers 3 and 5, but, of course only one of either of these first and second dielectric layers need be used.

[Effect of the Invention]

In the present invention, a cap, to the surface of which a hygroscopic material is attached, is affixed to a transparent substrate which forms an EL thin film layer, and an adhesive injection port is sealed with a thermosetting resin adhesive; by this structure, and furthermore, with sealing being performed under conditions in which heating is performed, a thin film EL element in which moisture prevention is at a high level can be obtained.

4. Brief Description of the Drawings

FIGS. 1(a) to 1(c), 2(a) and 2(b), and 3(a) to 3(c) are diagrams illustrating one embodiment of the invention. FIGS. 1(a) to 1(c) are cross-sectional view drawings showing steps of a sealing method. FIG. 2(a) is a plane view diagram illustrating a transparent substrate formed of a thin film layer, and FIG. 2(b) is a cross-sectional view diagram thereof. FIG. 3(a) is a plane view drawing illustrating a cap, FIG. 3(b) is a cross-sectional view diagram thereof taken along line b-b, and FIG. 3(c) is a perspective view diagram. FIG. 4(a) is a plane view diagram illustrating a different example of a structure of the cap, FIG. 4(b) is a cross-sectional view diagram thereof taken along line b-b, and FIG. 4(c) is a perspective view diagram. FIGS. 5(a) and 5(b), 6(a) and 6(b), 7(a) and 7(b), and 8(a) and 8(b) are diagrams illustrating steps of a sealing method which uses this cap, where each of FIGS. 5(a), 6(a), 7(a), and 8(a) is a cross-sectional view diagram and each of FIGS. 5(b), 6(b), 7(b), and 8(b) is a side view diagram taken from a direction differing by 90° from the direction in which FIGS. 5(a), 6(a), 7(a), and 8(a) are taken.

1....transparent substrate; 2....transparent electrode; 3, 5....dielectric layers; 4....EL light-emitting layer; 6....back surface electrode; 7....EL thin film layer; 8, 15....caps; 9....notch (adhesive injection port); 10....solidified adhesive layer; 11....hygroscopic particles; 13, 14, 21, 22....solidified adhesive joints; 16....slit (adhesive injection port).

Patent Applicant: Hoya Corporation

Representative: Patent Attorney, Masaki YAMAKAWA (and two others)

橙色の発光を呈する。

このようなEL素子は、透明基板上に形成した透明電極から背面電極に至る薄膜積層物（以下EL薄膜層という）が外気、特に湿気の影響を受けやすく、空気中の湿気がEL薄膜層にわずかに吸着しても、それが誘電体層等のピンホールなどに侵入してその部分の抵抗を下げる。その結果過大な電流が流れて局部的に発熱することになり、薄膜が基板から剥離したり、絶縁破壊を起こして素子寿命を低下させることになる。また、侵入した湿気がEL発光層まで到達すると、発光層は水に対してきわめて弱いために劣化して、素子寿命が低下することになる。

そこで、このような湿気から薄膜EL素子を保護するために、従来より、①；無機あるいは有機性の被覆でEL薄膜層全体を密着して覆う、②；ガラスキャップなどでカバーする、③；②のガラスキャップ中を脱ガス真空化する、④；②のガラスキャップ中にシリコンオイル等の絶縁性液体を満たす、⑤；④に加え、さらにキャップ内壁に水分吸収剤としてシリカゲルを塗布したシートを貼設するなどの対策が施されてきた。

〔発明が解決しようとする問題点〕

上述した各種の防湿対策は、いずれも効果上ならびに製造上の欠点を有する。まず、①はEL素子の放熱を行なうのに不都合で、場合によつては応力の増加による膜剥離が生ずる。次に、②は効果が不十分である。さらに、③，④，⑤はいずれも製造上の困難性を有し、また特に③は外部大気圧とキャップ内部との圧力差のために、素子強度の点でも問題がある。

〔問題点を解決するための手段〕

本発明による薄膜EL素子は、内面に吸湿性材料を固定したキャップを用い、かつこのキャップに設けた接着剤注入口を、熱硬化形樹脂接着剤により封じしたものである。

また本発明による製造方法は、キャップ内面に吸湿性材料を分散させた接着剤を塗布し硬化させて吸湿性材料を固定する工程と、EL薄膜層を形成した透明基板にキャップを固着する工程と、これらを加熱した状態でキャップの接着剤注入口に熱硬化形樹脂接着剤を注入し硬化させて封止する工程とを設けたものである。

〔作用〕

キャップを透明基板上に配置し加熱することにより、内部に残留する湿気が接着剤注入口から追い出されるとともに、外部雰囲気からの湿気の侵入が阻止される。封じ後、内部に少量の湿気が残留したとしても、その湿気はキャップ内面に固定された吸湿性材料により吸収される。

〔実施例〕

第2図は、透明基板上に形成したEL薄膜層を示し、同図(a)は平面図、同図(b)はそのb－b断面図である。次に、その形成方法を説明する。

まず、アルミノシリケートガラス（HOYA(株)製NA40；寸法100×100×3㎜）からなる透明基板1上に、スズ酸化物を混入した酸化インジウムを素材にして、真空蒸着法により多数の帯状の透明電極2を成膜する。このとき、透明電極2の引出し部は、交互に基板の周縁に向けて延在させる。

次に、同様の製法により、酸化イットリウム（Y_2O_3）を素材にして第1の誘電体層3（膜厚3000Å）、活性物質として0.5wt%のマンガン（Mn）をドープした$ZnS:Mn$焼結ペレットを素材にしてEL発光層4（膜厚6000Å）および第1の誘電体層3と同様な物質を素材にして第2の誘電体層5（膜厚3000Å）を順次成膜する。次いで第2の誘電体層5の上に、同様な製法により、アルミニウム（Al）を素材にして、透明電極2と互いに交差する方向に、多数の帯状の背面電極6（膜厚3000Å）を成膜する。この背面電極6の引出し部も、基板の周縁に向けて交互に延在させる。引出し部を除いた両電極ならびにその間に介在させたEL発光層および第1、第2の誘電体層により、EL薄膜層7が構成される。

第3図は、この透明基板1に固着して用いるキャップを示し、同図(a)は平面図、同図(b)はそのb－b断面図、同図(c)は斜視図（ただし裏返し）である。キャップ8は、透明基板1と同様の素材であるアルミノシリケートガラスからなる。各部の

寸法は第2図中に示した通り（単位はmm）で、概ね正方形の底壁および4つの側壁を有する箱形であるが、開放端部に、矩形状の切欠9を設けてある。この切欠9は、後述するように、キャップ8が透明基板5上に固着されたときに接着剤注入口を構成する。

次に、第1図を用いて、このようなキャップ8を透明基板1に固着して封じする工程を説明する。

はじめに、キャップ8を、100～200℃（本実施例では150℃）で1時間以上（同2時間）予備加熱し、表面に吸着された湿気を十分に除去する。次に、五酸化二リン（P_2O_5）の微粒子を熱硬化形のエポキシ系接着剤に分散させたペーストを準備し、キャップ8のEL薄膜層7に面することになる底壁内面に、、同ペーストをスクリーン印刷法により約200～300μmの厚さに塗布した後、ホットプレート等の加熱器に乗せ、100～200℃（本実施例では150℃）に加熱する。これにより、固化した接着層10に五酸化二リンの微粒子11が固定された吸湿効果層12が得られる（第1図(a)）。

一方、EL薄膜層7を形成した透明基板1を、100～200℃（本実施例では120℃）で1時間以上（同3時間）予備加熱して表面に吸着されたり内部に吸蔵されたりした湿気を十分に除去する。引続きさらに加熱しながら、この透明基板1上に、前述したキャップ8を、EL薄膜層7を覆うような所定位置に配置する。このとき、キャップ8の切欠9を除く開放端面に、予め熱硬化形のエポキシ系接着剤を塗布しておくことにより、その接着剤が硬化して形成された固化接着部13によつて、キャップ8は透明基板1上に固着される（第1図(b)）。

次に、これら透明基板1およびキャップ8の加熱をしばらく継続した後、その状態で、キャップ8の切欠9に新たなエポキシ系接着剤を注射器等で注入し、所定時間（本実施例では1時間）加熱し続ける。これにより、切欠9は、固化接着部14により封じされる（第1図(c)）。

封じに際し、キャップ8およびEL薄膜層7が

形成された透明基板1をそれぞれ予備加熱することによりそれらの表面に吸着された湿気あるいはEL薄膜層7中に吸蔵された水分を除去することができ、さらに両者を重ねた状態で加熱することにより、内部に残留する湿気をキャップ8の切欠9から外部に追い出すことができるとともに、外部雰囲気からの湿気の侵入を阻止することができる。また、封じ完了後に内部に少量の湿気が残留したとしても、その湿気は五酸化二リンの微粒子11により吸収されるため、EL薄膜層7が劣化するなど発光表示に悪影響が及ぶことを防止できる。この五酸化二リンによる吸湿効果は、封じの際に加熱されることから、さらに高められる。また、この五酸化二リンの微粒子11を固定している接着剤は熱硬化形であることから、封じ工程における加熱によつて劣化することはない。

本実施例において、透明基板1およびキャップ8の材質をともにアルミノシリケートガラスとしたが、これに限定されるものではない。例えばセラミックスやプラスチックでもよいが、好ましくは熱膨張係数の等しいガラスを用いることがよい。このようなガラスとしては、例えばソーダライムガラス等の多成分系ガラスでもよいし、石英ガラスでもよい。

また、熱硬化形樹脂接着剤は、エポキシ樹脂接着剤に限らず、シリコーン樹脂など、他の耐熱性・耐湿性の樹脂でもよい。

さらに、吸湿性材料としては、五酸化二リンの他に、シリカゲル、塩素酸マグネシウム（$Mg(ClO_3)_2$）、酸化アルミニウム（Al_2O_3）、酸化カルシウム（CaO）などを用いてもよい。

なお、矩形状の切欠9の代りに、V字状、U字状等の切欠または円管状、角管状等の貫通孔でもよく、その数は1対に限らず、少なくとも1個あればよい。また、貫通孔状の接着剤注入口であれば、キャップ8の側壁に限らず、底部に設けてもよい。

さらに、上述した実施例では、底部および側壁部を一体に形成したキャップを用いたが、これらを別体に形成して組合せてもよい。第4図に、そ

の例を示す。第４図(a)は平面図、同図(b)はそのｂ－ｂ断面図、同図(c)は斜視図である。

本実施例のキャップ１５は、透明基板１と同一のアルミノシリケートガラスからなる板状カバー15Aの一主表面に、コの字形の枠状のスペーサ15Bを２個向かい合せに配置した構成を有している。スペーサ15Bは、板状カバー15Aに熱硬化形エポキシ系接着剤を塗布し、オーブン等で100℃で加熱硬化させることにより固着形成した。その際、２個のスペーサ15Bのコの字の開口側を相互に所定距離Ｌ＝0.3～10㎜（本実施例では３㎜）だけ離間させ、スリット１６を構成する。なお、２個のスペーサの枠内は、ＥＬ薄膜層７の側面を包囲する大きさを有する。また、その幅寸法Ｗは0.5～５㎜（本実施例では２㎜）が適当である。高さ寸法Ｈは、ＥＬ薄膜層７のうちの透明電極２を除いた全膜厚と、後述する固化吸着層の厚さ（通常20～3000μm；本実施例では200～300μm）とを合せた厚さより大きければよく、本実施例では1000μmとした。

次いで、スペーサ15Bの外側面１７と板状カバー15Aおよび透明基板１の対向面１８，１９とで構成される凹所２０に、新たなエポキシ系接着剤を注射器等で注入し、所定時間（本実施例では１時間）加熱を続ける。これにより、エポキシ系接着剤が硬化し、固化接着部２１を形成する（第７図）。固化接着部２１は、透明基板１、板状カバー15Aおよびスペーサ15Bの外側面に固着し、これらを強固に接合している。図では、誇張して描いてあるが、固化接着部２１の高さ寸法（スペーサ15Bの高さ寸法と同じ）Ｈは、前述した通り1000μm（１㎜）にすぎないから、透明基板１と板状カバー15Aとの間における表面張力を得て、接着剤は容易に注入される。なお、このときスリット１６には接着剤は注入されず、内部空間と外部雰囲気とはスリット１６により通じている。

この状態で、透明基板１およびキャップ１５の加熱をしばらく継続した後、スリット１６に新たなエポキシ系接着剤を注射器等で注入し、所定時間（本実施例では２０分間）加熱し続ける。これにより、スリット１６は固化接着部２２により封止される（第８図）。

次に、第５図ないし第８図を用いて、このようなキャップ１５を透明基板１に固着して封じする工程を説明する。なお、各図において(a)は第４図(b)に対応する断面図、(b)は側面図である。

はじめに、キャップ１５を100～200℃（本実施例では120℃）で１時間以上（本実施例では２時間）予備加熱し、表面に吸着されたり内部に吸蔵された湿気を十分に除去する。次に、五酸化二リン（P_2O_5）の微粒子を熱硬化形のエポキシ系接着剤に分散させたペーストを、第１図の実施例と同様の方法により、板状カバー15AのＥＬ薄膜層７に面する内面に20～3000μm（本実施例では200～300μm）の厚さに塗布し、硬化させて、五酸化二リンの微粒子１１と固化した接着層１０とからなる吸湿効果層１２を得る（第５図）。

一方、ＥＬ薄膜層７を形成した透明基板１も、第１図の実施例と同様に加熱し、湿気を十分に除去した後、引続き100～200℃（本実施例では150℃）で加熱しながら、キャップ１５を、ＥＬ薄膜層７を覆う所定位置に配置する（第６図）。

本実施例において、透明基板１、板状カバー15A、熱硬化形樹脂接着剤および吸湿性材料などが上述した物質に限定されないことは、第１図の実施例の場合と同様である。

また、各実施例において、吸湿性材料はキャップの底部内面（第５図の実施例では板状カバー15Aの内面）にのみ固定したが、キャップの側壁内面（第５図の実施例ではスペーサ15Bの内面）に固定してもよく、キャップ内面全体に固定すればその吸湿効果をより高めることができることはいうまでもない。

なお、ＥＬ発光層４を第１および第２の誘電体層３，５で挟んだ構成を示したが、これら第１および第２の誘電体層は、いずれか一方のみでもよいことはいうまでもない。

〔発明の効果〕

本発明によれば、内面に吸湿性材料を固定したキャップを、ＥＬ薄膜層を形成した透明基板に固

着し、接着剤注入口を熱硬化形樹脂接着剤で封じした構成をとることにより、またその封じを加熱した状態で行なうことにより、防湿効果の高い薄膜EL素子が容易に得られる。

4. 図面の簡単な説明

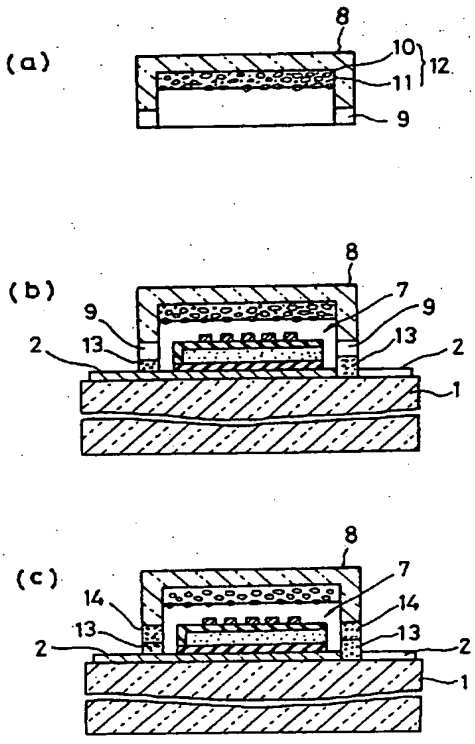
第1図ないし第3図は本発明の一実施例を示す図で、第1図は封じ方法を示す工程断面図、第2図(a)はEL薄膜層を形成した透明基板を示す平面図、同図(b)はその断面図、第3図(a)はキャップを示す平面図、同図(b)はそのb－b断面図、同図(c)は斜視図、第4図(a)はキャップの他の構成例を示す平面図、同図(b)はそのb－b断面図、同図(c)は斜視図、第5図～第8図はこのキャップを用いての封じ方法を示す工程図で、各図(a)は断面図、各図(b)は(a)と90°方向の異なる側面図である。

1・・・・透明基板、2・・・・透明電極、3，5・・・・誘電体層、4・・・・EL発光層、6・・・・背面電極、7・・・・EL薄膜層、8，15・・・・キャップ、9・・・・切欠(接着剤注入口)、10・・・・固化接着層、11・・・・吸湿性物質の微粒子、13，14，21，22・・・・固化接着部、16・・・・スリット(接着剤注入口)。

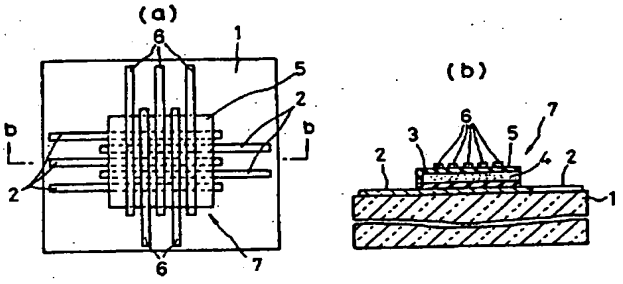
特許出願人　ホーヤ株式会社

代理人　山川政樹(ほか2名)

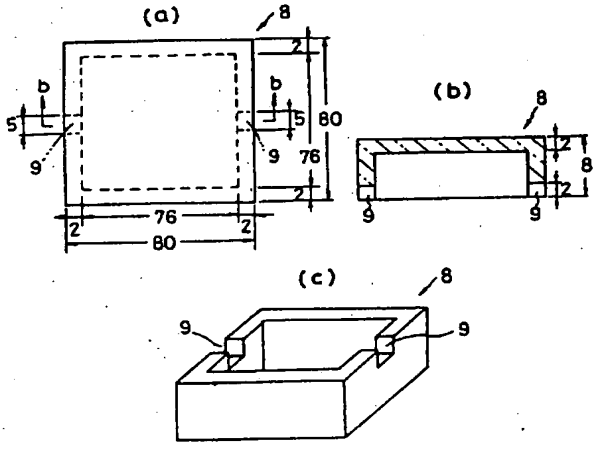
第1図



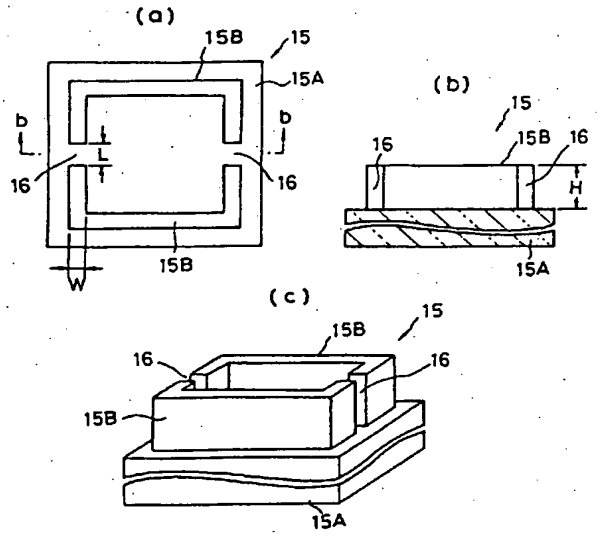
第2図



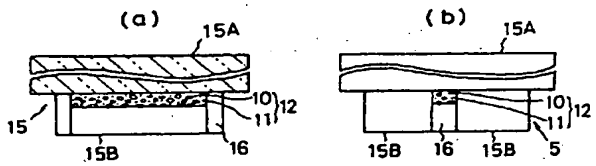
第3図



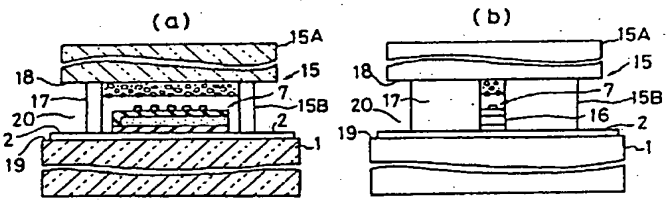
第 4 図



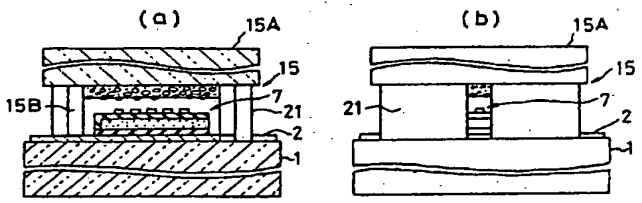
第 5 図



第 6 図



第 7 図



第 8 図